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FINAL REPORT

REPORT NO. 16898-6009-R000

STUDY OF THE FORMATION AND BEHAVIOR OF CLOGGING MATERIAL IN PROPELLANTS

(OXYGEN DIFLUORIDE AND DIBORANE)

JANUARY 1971

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PREPARED FOR

THE JET PROPULSION LABORATORY
PASADENA, CALIFORNIA 91103

UNDER

NATIONAL AERONAUTICS AND SPACE
ADMINISTRATION CONTRACT NAS 7-787

TRW
SYSTEMS GROUP

ONE SPACE PARK • REDONDO BEACH • CALIFORNIA

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FOREWORD

This final report was prepared by TRW Systems Group, Redondo Beach, California. It contains the results of work accomplished during the period April 13, 1970 to January 13, 1971, under NASA Contract NAS 7-787, titled "Study of the Formation and Behavior of Clogging Material in Propellants." The propellants, liquid diborane and liquid oxygen difluoride, studied under this program were flowed through small constrictions to determine if flow clogging could be partially or fully initiated. The program was originated and technical management provided by the Jet Propulsion Laboratory, Pasadena, California, under the direction of Mr. Louis R. Toth. The NASA Program Manager was Mr. Frank E. Compitello, Code RPL, Office of Advanced Research and Technology; National Aeronautics and Space Administration, Washington D. C.

The work performed on the program was accomplished by the Science and Technology Division of TRW Systems Group. Technical direction of the program was provided by the Chemical Propulsion Department of the Combustion Systems Laboratory. The program manager was Mr. R. J. Salvinski. Responsible technical personnel were Dr. E. A. Burns, Mr. J. R. Denson and Dr. J. Neff of the Chemical Research and Services Department, Mr. J. L. Reger of the Materials Science Department, and Mr. M. J. Makowski of the Chemical Propulsion Department. Mr. C. E. Armstrong of the Product Design and Analysis Department was responsible for instrumentation.

CONTENTS

		Page
1.0	INTRODUCTION AND SUMMARY	1-1
	1.1 Purpose and Scope	1-2
	1.2 Experimental Study	1-2
	1.2.1 Propellant Flow Testing	1-2
	1.3 Specific Tasks	1-2
	1.4 Summary and Conclusions	1-4
	1.4.1 Diborane Propellant Flow Tests	1-4
	1.4.2 Oxygen Difluoride Propellant Flow Tests	1-4
2.0	PROPELLANT FLOW SYSTEM	2-1
	2.1 Flow System Design	2-1
	2.2 Supply Tank Design	2-1
	2.3 Heat Exchanger Design	2-5
	2.4 Description of the Test Sections	2-5
3.0	DIBORANE FLOW STUDY	3-1
	3.1 Introduction	3-1
	3.2 Characterization of Propellants	3-1
	3.2.1 B_2H_6 Doping Experiments	3-4
	3.2.2 Analysis of Diborane	3-6
	3.3 Diborane Flow Test Plan	3-7
	3.4 Flow Bench Cleaning Procedure	3-10
	3.5 Leakage Check Procedures	3-10
	3.6 Diborane Flow Test Results	3-10
4.0	OXYGEN DIFLUORIDE FLOW STUDY	4-1
	4.1 Introduction	4-1
	4.2 Characterization of Propellants	4-1
	4.2.1 OF_2 Propellant Dopants	4-2
	4.3 Test Plan	4-3
	4.4 Cleaning Procedure	4-5
	4.5 Leakage Procedure	4-5
	4.6 OF_2 Flow Test Results	4-5

1.0 INTRODUCTION AND SUMMARY

This report represents the results of the work performed under NASA Contract NAS 7-787 to investigate the behavior of diborane and oxygen difluoride propellant flow through small constrictions. Earlier work[1,2] in which nitrogen tetroxide was flowed through small constrictions showed the propellant to form gel-like materials at the entrance of the constriction resulting in flow decay and, in many cases, complete blockage. The nitrogen tetroxide clogging material was identified as a gel which formed a powdery residue upon drying in air. The powder contained iron which was believed to be the impurity which acted as a nucleation site for the precipitation of the gel-like material.

Additional work was performed flowing hydrazine propellant through capillaries.[3] No apparent flow anomalies were found attributable to the formation of gel or viscous material. Several runs flowing neat diborane and neat oxygen difluoride through a 0.010 inch diameter capillary were also made.[3] No evidence of flow anomalies were found.

This program was designed to continue the earlier efforts with diborane and oxygen difluoride to determine the effects of temperature conditioning and propellant aging on these propellants flowing through orifices, filters and acquisition screens. The propellants were artificially doped and were cooled prior to entering the test section. The propellant flow test conditions were designed to represent or exceed the velocity and temperature extremes that would be experienced under actual space flight conditions. A summary of the contractual requirements are presented in the following sections.

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(1) "Investigation of the Formation and Behavior of Clogging Materials in Earth and Space Storable Propellants," Interim Report 08113-6007-R000, October 1967.

(2) "Investigation of the Formation and Behavior of Clogging Materials in Earth and Space Storable Propellants," Interim Report 08113-6016-R000, October 1968.

(3) "Investigation of the Formation and Behavior of Clogging Materials in Earth and Space Storable Propellants," Final Report 08113-6025-R000, November 1969.

1.1 PURPOSE AND SCOPE

It was the purpose of this program to investigate liquid propellant clogging behavior under varying flow conditions and to determine the formation of clogging material and its effect on the behavior of the propellant flow.

Emphasis was to be placed on the characterization and behavior under flow conditions of the space storable propellants oxygen difluoride (OF_2) and diborane (B_2H_6).

The scope of the effort for investigation of liquid propellant clogging behavior of oxygen difluoride (OF_2) and diborane (B_2H_6) included the inclusion of small constriction type elements in the flow studies. Neat and doped propellants were to be flowed and the characterization of the propellants were to be determined.

1.2 EXPERIMENTAL STUDY

The laboratory testing concentrated on the flow behavior and characterization of oxygen difluoride (OF_2) and diborane (B_2H_6).

Design parameters considered are typical of or scalable to those in real systems and components.

1.2.1 Propellant Flow Testing

Flow testing was performed to determine the extent and behavior of flow degradation and clogging of small clearances under propellant flow conditions. Oxygen difluoride and diborane were flowed through selected components under conditions of varying temperature and velocity. These components included a filter, acquisition screens and an orifice test section; the measurements made during flow were pressure, temperature and flow rate.

1.3 SPECIFIC TASKS

The summary of specific tasks accomplished on this program is presented in Table 1-1.

Table 1-1. Summary of Specific Tasks

NAS 7-787 Study of the Formation and Behavior of Clogging Material in Propellants

Period April 1970 to December 1970

OBJECTIVE: Determine the flow behavior of B_2H_6 and OF_2 propellants flowing through small constrictions.

1. B_2H_6/OF_2 FLOW SYSTEM - Design and Fabricate Flow Bench for B_2H_6 and OF_2 Service

   A. Closed Loop System
   B. All Metal System
   C. Positive Expulsion
   D. Temperature Conditioning
   E. Test Sections
      a) .010" and .020" Orifice
      b) 2μ Nominal - 10μ Absolute
      c) 10μ Absolute Acquisition Screen
   F. Instrumentation: ΔP, T, $\dot{w}$

2. B_2H_6 FLOW TESTS - Flow B_2H_6 Through Small Constrictions to Determine Flow Behavior.

   A. B_2H_6 Propellant Characterization
   B. Neat B_2H_6
   C. Doped B_2H_6
      a) Stainless Steel Oxides
      b) Aluminum Oxides
      c) Petroleum Ether
      d) Teflon T-5B DuPont Resin
   D. Test Sections
      a) .010" Diameter Orifice
      b) .020" Diameter Orifice
      c) 2μ Nominal - 10μ Absolute
      d) 10μ Absolute Acquisition Screen
   E. Temperature Conditioned
      -60°F to -240°F

3. OF_2 FLOW TESTS - Flow OF_2 Through Small Constrictions to Determine Flow Behavior.

   A. OF_2 Propellant Characterization
   B. Neat OF_2
   C. Doped OF_2
      a) Stainless Steel Oxides
      b) Aluminum Oxides
   D. Test Sections
      a) .010" Diameter Orifice
      b) .020" Diameter Orifice
      c) 2μ Nominal - 10μ Absolute
      d) 10μ Absolute Acquisition Screen
   E. Temperature Conditioned
      -180°F to -240°F

1.4 SUMMARY AND CONCLUSIONS

1.4.1 Diborane Propellant Flow Tests

Approximately one hundred flow tests were conducted flowing liquid diborane through a 0.010" diameter and a 0.020" diameter orifice, a 2μ nominal - 10μ absolute rated filter and a 10μ absolute rated acquisition screen. The diborane propellant used was both neat and doped and flowed under both isothermal and changing temperature (chilling) conditions. The dopants used were oxides of stainless steel, oxides of aluminum, low molecular weight hydrocarbons and finely divided Teflon. Temperature was varied from -60°F to -240°F for several experiments.

No evidence of flow decay or blockage of any test section was found. Flow variations as high as 20% within a single run were noted for several runs where temperature conditioning was employed. The probable cause of these variations is believed to be changes in viscosity of the diborane. For the average test run flow variations were within 10%. However, the variations occurred over short durations of the total run time and no evidence or trends could be established indicative of flow decay or flow clogging. Within the test conditions reported it is concluded that diborane does not exhibit flow anomalies caused by the formation of clogging materials or any polymorphic changes inherent in the liquid diborane.

1.4.2 Oxygen Difluoride Propellant Flow Tests

Liquid oxygen difluoride flowed through a 0.010" diameter orifice, a 2-10μ filter and a 10μ acquisition screen showed no evidence of flow clogging or flow decay. The propellant was run both neat and doped for the orifice and filter test runs. Neat propellant was flowed through the acquisition screen. Temperature conditioned runs ranged from -180°F to -240°F. Approximately a 10% variation in flow was noted during a single run for several runs. Some difficulty was experienced in controlling flow. In several runs flow rate increased and then decreased and increased again indicating erratic flow control. The flow control may be inherent in the flow bench or instrumentation. Temperature variations may have been responsible causing changes in density and viscosity. However, in all cases, no trends were apparent which indicated flow clogging or flow decay behavior of oxygen difluoride.

2.0 PROPELLANT FLOW SYSTEM

2.1 FLOW SYSTEM DESIGN

The flow bench system schematic is shown in Figure 2-1. The flow bench system depicted in Figure 2-1 was installed in a temperature controlled test chamber. Figures 2-2 and 2-3 are photographs of the flow system hardware and the test chamber. Operating conditions and bench components are summarized under Table 2-1. The test chamber was cooled by flowing chilled nitrogen through two twelve-inch, high speed fans causing the nitrogen to disperse throughout the chamber. This allowed the flow bench system to be uniformly cooled to the desired operating temperatures.

The flow bench was designed to include a heat exchanger to temperature condition the propellants during flow. The flow bench is an all metal system and no polymers are used where propellants contact the system. The supply tank consists of a bellows pressurized externally with helium. The propellant expelled from the bellows is flowed through the heat exchanger prior to entering the test section. After leaving the test section the propellant is fed to a receiver tank. The receiver tank is a pressurized bellows identical to the supply tank. A detailed description of the supply tank, heat exchanger and test section is given in the following sections.

2.2 SUPPLY TANK DESIGN

The propellant supply tank consists of a calibrated bellows type accumulator. An identical accumulator is used for the receiver. This allows a completely closed propellant system during testing. The bellows is an edge welded convoluted design and is fabricated of 347 stainless steel. A rod is provided at the moving bellows head to allow attachment of the LVDT position monitoring device. The bellows stroke is 10 inches for an expulsion of approximately 1 liter of propellant. Cavities are provided in the accumulator base to hold three electrical heaters. These heaters were used to temperature condition the propellant in the supply tank to the desired value before starting a flow run.

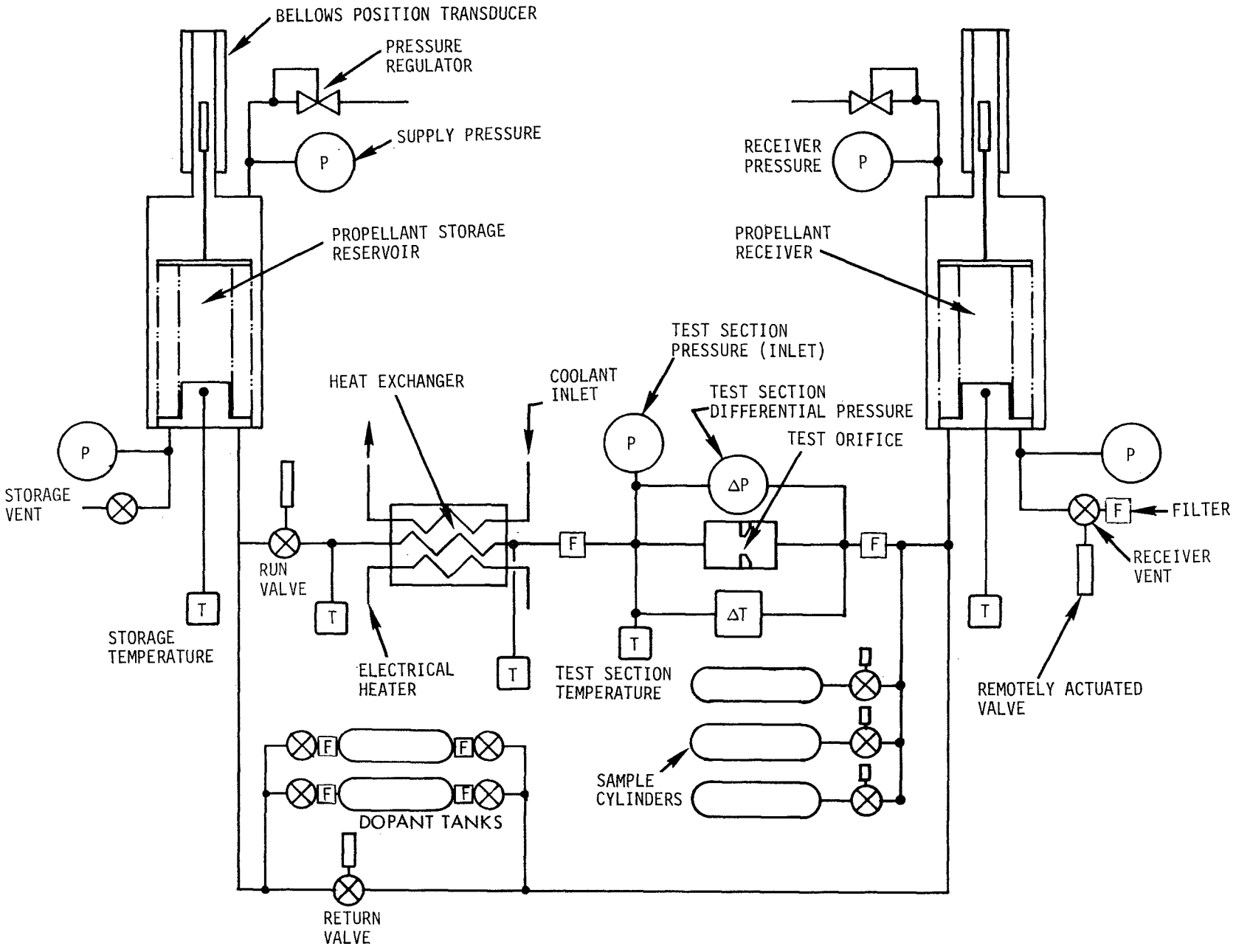

Figure 2-1. Flow System Schematic

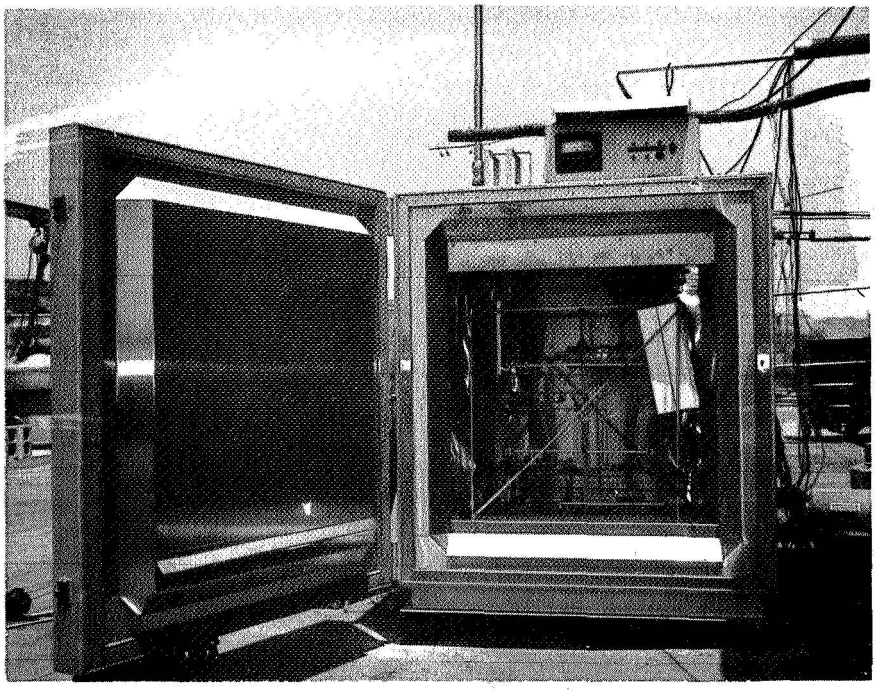

Figure 2-2. Flow System Mounted in the Environmental Chamber

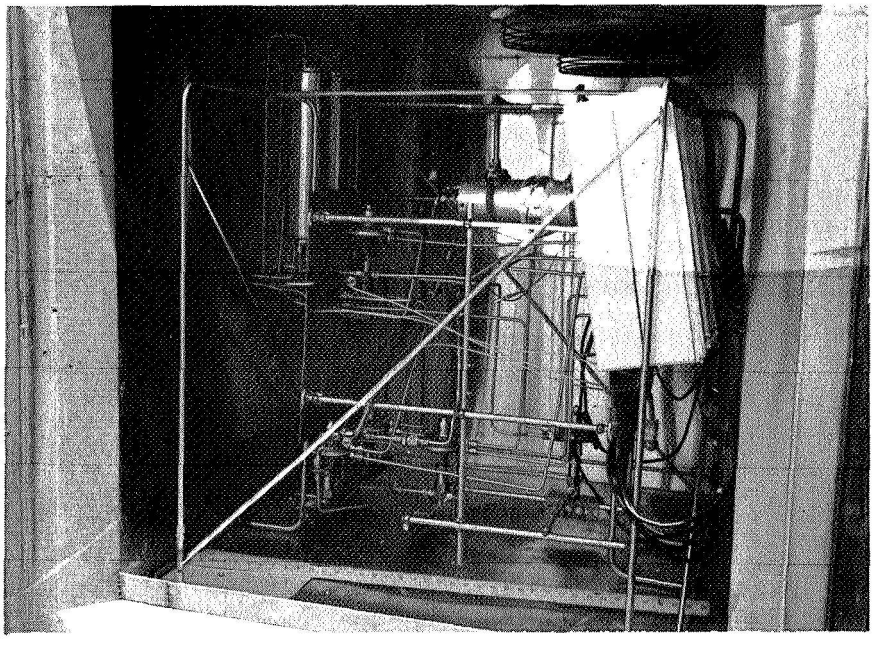

Figure 2-3. Closeup View of Flow System

Table 2-1. Operation Conditions and Bench Components for Flow Decay Studies

Environmental Test Chamber Bemco Inc.	40 cu. ft. box
Temperature	-140°C
Expected Temperature Control	± 4° at -140°C
Pressurant Gas Range	>300 psig
Pressurant Gas	Helium
Maximum Operating Pressure	300 psi
Propellant Holding Tank	
Capacity	1 liter
Material	Stainless steel
Receiver Tank	
Capacity	1 liter
Material	Stainless steel
Remote Control Valve	1/4 in. Hoke 477 series bellows valve; copper seats
Hand Operated Valves	Hoke 4214 bellows valve; 305 stainless steel seats
Line Tubing	304 series 1/4 in. x .028 in. wall, 305 stainless steel AN flared fittings with 305 stainless steel conical seal.
Line Filters	1/2 in. AN fitting in-line filters, wire mesh 316 stainless steel
Pressure Transducers	Taber Teledyne, N. Tonawanda, N.Y. 0-300 psi range, Model 254

2.3 HEAT EXCHANGER DESIGN

A heat exchanger was designed to temperature condition the propellant flow from the storage reservoir temperature to the desired test section temperature. Electrical heaters were used for temperature control. These heaters allow any desired propellant outlet temperature, to the maximum cooling limits of the exchanger to be preset and maintained before propellant flow is initiated as well as during a flow run.

The heat exchanger is used as follows: The propellant is contained in a 0.125 inch stainless steel diameter tube which is helically wound into grooves in the exchanger's central mandrel. Cooling is provided by a counterflow of cold N_2 gas and/or liquid nitrogen. The liquid nitrogen flows in helical channels and is vaporized and heated by electrical heaters. The heater power is controlled to maintain the propellant outlet temperature at the desired level. An outer cylinder contains the N_2 flow and forces effective heat transfer between the N_2 and the 0.125 inch diameter propellant tube.

2.4 DESCRIPTION OF THE TEST SECTIONS

The test section was designed to allow changing orifice sizes and filters and screen with relative ease and to hold the output temperature and pressure instrumentation ports. A cross-sectional view is shown in Figure 2-4. The test section body is manufactured from a 1/4-inch tube size AN cross. The body and inserts are 304 CRES. The outlet thermowell sensing area is placed in the outlet jet of the orifice to allow measurement of outlet fluid temperature. The pressure and temperature taps were placed one pipe diameter downstream of the orifice. This is one of the standard measurement locations used in orifice flowmeters.

Pressure transducers were isolated from the ambient conditions in the environmental test chamber by an insulated box held at -60°F. This allowed the transducers to operate at a temperature within the manufacturer's specifications but at a temperature low enough to prevent pyrolysis of the diborane.

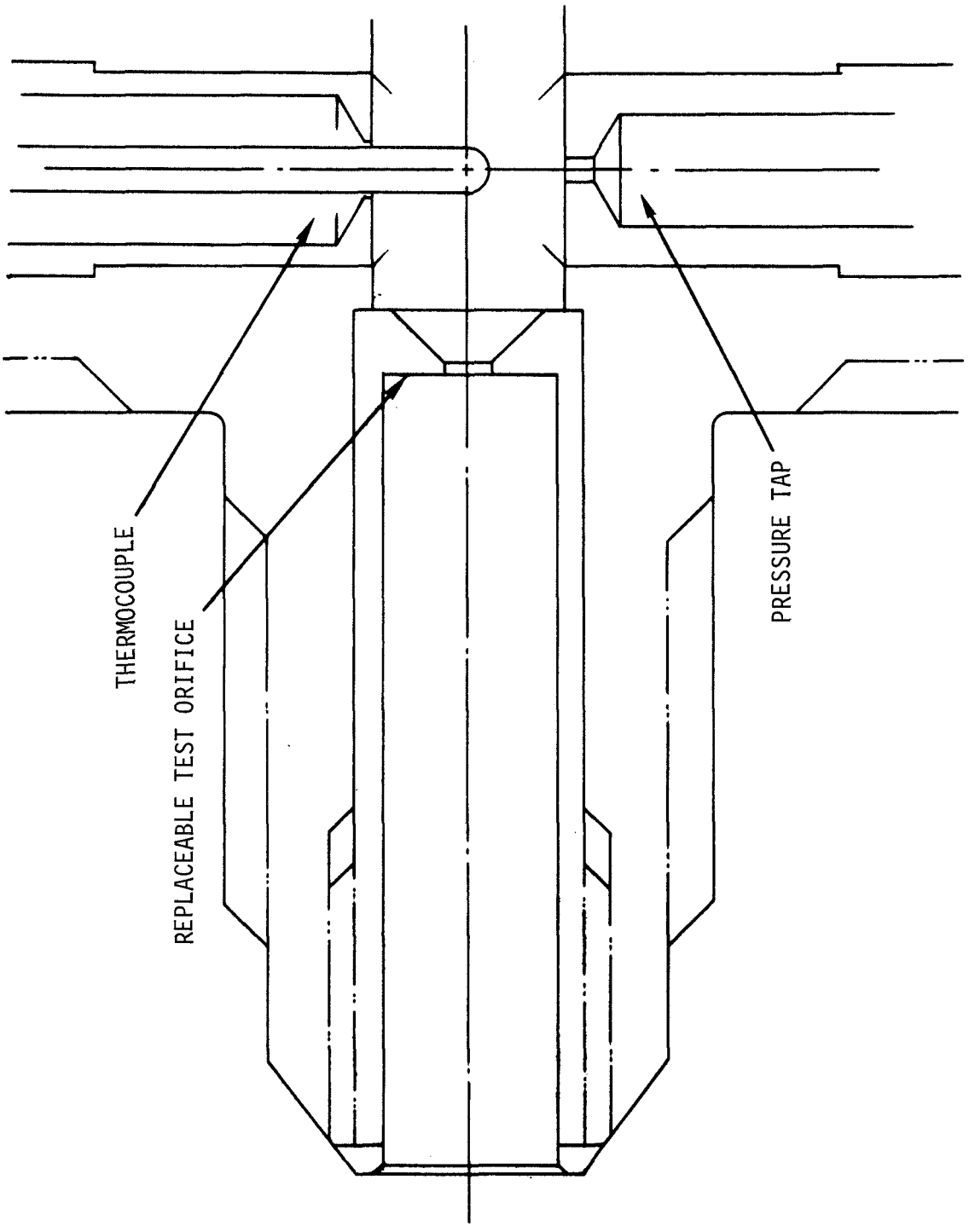

Figure 2-4. Flow Test Section - Orifice (10X Size)

Figures 2-5 through 2-7 show the detailed test sections used in the program. The 2-10μ filter was purchased from the Western Filter Co. P/N S4-19310-2, and designed to be installed in a 1/4 inch AN fitting. The acquisition screen was a twilled dutch weave type 304L, 375 x 2300 mesh, 10μ absolute rating.

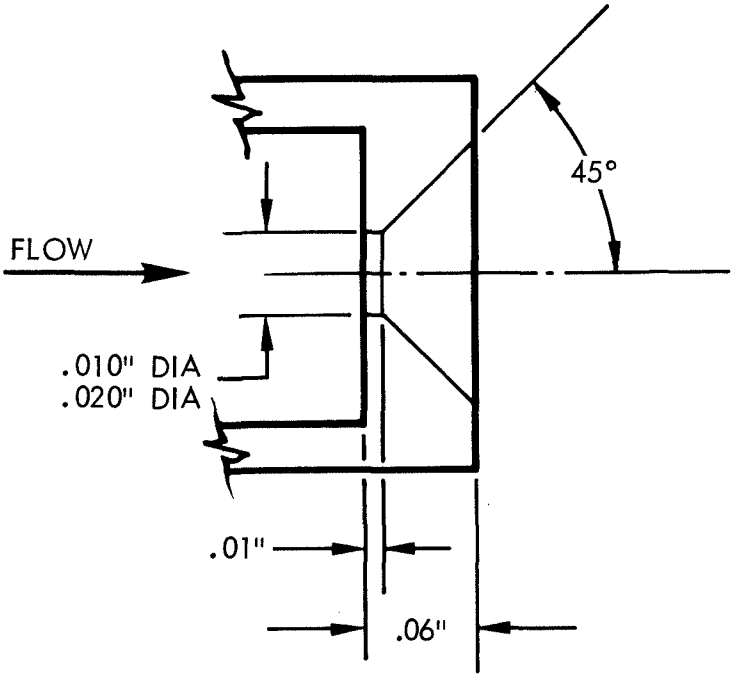

Figure 2-5. Orifice Test Section

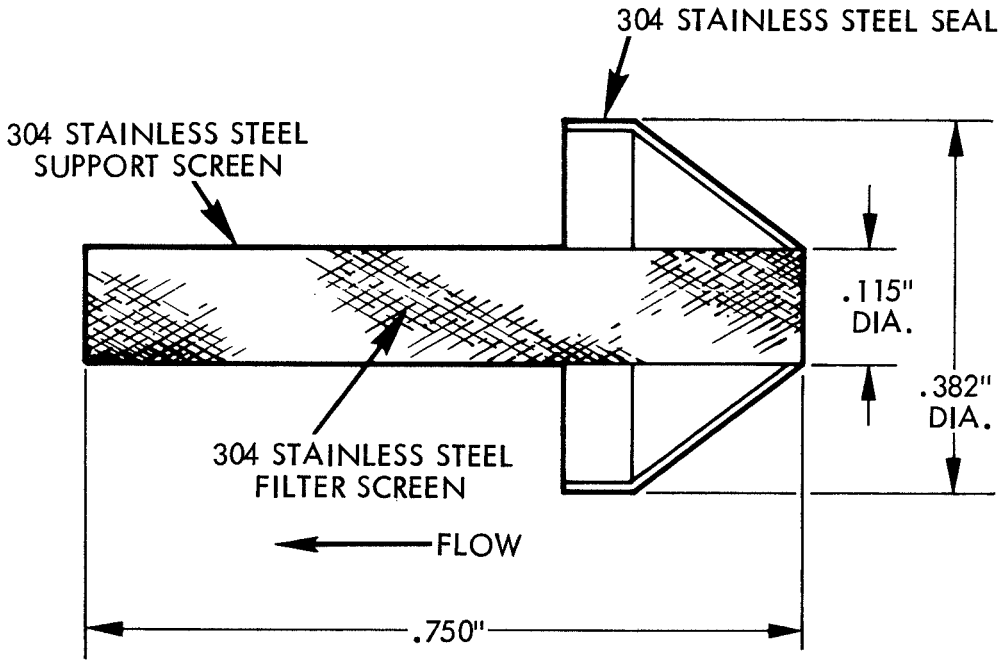

Figure 2-6. 2-10μ Filter Test Section

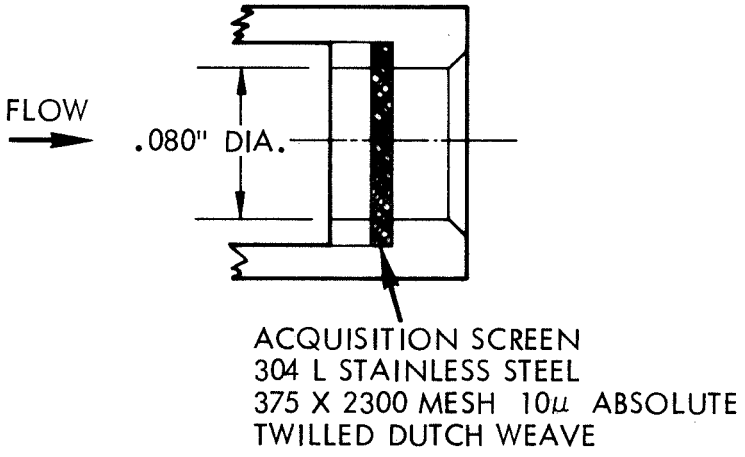

Figure 2-7. 10μ Acquisition Screen Test Section

3.0 DIBORANE FLOW STUDY

3.1 INTRODUCTION

The scope of the effort to investigate liquid B_2H_6 propellant clogging behavior included determining the flow characteristics of liquid diborane through orifices, screens and filters. Approximately one hundred experiments were conducted under conditions of changing temperature, velocity, and propellant. Temperature conditioning of the liquid diborane during the flow experiments included experiments in which diborane was chilled in a heat exchanger in the range from -60°F to -240°F to determine whether possible precipitates initiated by chilling contribute to any flow anomalies.

Neat and doped diborane were flowed through orifices and a filter. Neat diborane was flowed through the acquisition screen.

3.2 CHARACTERIZATION OF PROPELLANTS

B_2H_6 is made commercially by the reduction of boron halides (BCl_3 or BF_3) or trimethyl borate [$B(OCH_3)_3$]. This reduction is accomplished with metal hydrides, sodium hydride (NaH) and lithium hydride (LiH). Intermediate complex hydrides are also involved in the halide synthesis. For example, sodium borohydride ($NaBH_4$) and lithium borohydride ($LiBH_4$) are intermediates in these syntheses. Solvents are hydrocarbon ethers; diethyl ether, dimethyl ether of ethylene glycol ($CH_3OCH_2CH_2OCH_3$) and dimethyl either of diethylene glycol [$CH_3O(CH_2CH_2O)_2CH_3$].

Most of the reactants, products and solvents, except the boron halides, are much less volatile than B_2H_6 and can readily be separated from B_2H_6 by simple fractionation. The boron halides are very unstable in the presence of the excess metal hydrides used in the syntheses and are completely reacted. The ethers, however, are sometimes reduced to saturated hydrocarbons, methane and ethane, and are trace contaminants in B_2H_6.

The major producer of B_2H_6, Callery Chemical Corporation, now uses the halide method of producing small quantities of B_2H_6[1] (quantities of 200 pounds) and will use the other method, reduction of trimethyl borate, for tonnage quantities. Therefore, impurities are likely to be different if the demand for B_2H_6 changes. Purity of presently delivered materials is guaranteed to be 96% or better (B_2H_6 liquid expanded to the gas phase) with the major impurity being H_2 gas and traces of hydrocarbons. It is not unusual to obtain B_2H_6 of 99+% purity. This wide range of purity is possible because B_2H_6, if allowed to contact surfaces near ambient temperature, can decompose to produce hydrogen and higher molecular weight and less volatile boron hydrides; the most common being tetraborane, B_4H_{10}, which can further react to form higher boron hydrides and hydrogen.

To prevent the formation of higher molecular weight pyrolysis products, B_2H_6, after preparation, is stored in mild steel tanks below 0°C in quantities of 200 pounds. Samples prepared for customers are removed from the storage tank by first blowing off volatile impurities, H_2 and methane, and transferring the B_2H_6 to dry ice chilled tanks (1 pound or 40 pounds). These steel tanks are shipped to the customer in dry ice coolant. This vapor transfer to the customer's tank removes most non-volatile impurities and usually only the most volatile hydrocarbons, methane, ethane and hydrogen, remain in the shipped B_2H_6.

The shipping tanks are a source of some impurities and the final purity of B_2H_6, as used, will depend upon the method of removing B_2H_6 from these tanks. A vapor transfer would again remove most non-volatile impurities; but if B_2H_6 is transferred as a liquid, the non-volatile impurities will not be removed.

Tanks are cleaned when necessary by a process that includes a hydrocarbon wash (high purity petroleum ether), steam cleaning, and vacuum drying.* *

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[1] Mr. Toering, "Private Communications," November 1969 and June 1970.

*Halogenated solvents (freons) for cleaning are avoided because of shock sensitivity with B_2H_6.

This would introduce petroleum ether contaminants, metal oxides, and water. Unless the delivery tank is sampled as a vapor rather than as a liquid, these contaminants remain and could cause blockage in small orifices.

The reaction of metal oxides in the storage tank with B_2H_6, a strong reducing agent, has been observed[2] with stainless steel oxides. These and the more noble metal oxides have always been considered potentially dangerous sources of exothermic reactions of B_2H_6 with the oxygen, as shown with a metal oxide, MO, in Equation (1).

$$3\ MO + B_2H_6 \longrightarrow B_2O_3 + 3H_2 + 3M \qquad (1)$$

or with metal hydroxides as shown in Equation (2).

$$3\ M(OH)_2 + B_2H_6 \longrightarrow 3M + 2B(OH)_3 + 3H_2 \qquad (2)$$

Other possible reactions in the tanks include the reaction of trace quantities of water with B_2H_6 to produce boric acid as shown in Equation (3), or O_2 with B_2H_6 to produce boric acid as shown in Equation (4).

$$3HOH + B_2H_6 \longrightarrow 2B(OH)_3 + 3H_2 \qquad (3)$$

$$B_2H_6 + 3O_2 \longrightarrow B_2O_3 + 3H_2O \longrightarrow 2B(OH)_3 \qquad (4)$$

Equations (1) and (3) probably account for most of the hydrogen in tanks kept at temperatures below -20°C for periods of 1-12 months.

Production of high molecular weight boron hydrides in chilled B_2H_6 is a slow process and has not been properly evaluated. The conventional yellow solids $(BH)_x$ produced by high temperature pyrolysis of B_2H_6 could cause difficulty if present, but the formation of these solids is assumed to be minimal in equipment used under normal conditions for storage of liquid B_2H_6.

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(2) "Investigation of the Formation and Behavior of Clogging Material in Earth and Space Storable Propellants," Final Report 08113-6025-R000, November 1969.

Summarizing, the impurities believed present and likely contributors to clogging in B_2H_6 propulsion systems would be low molecular hydrocarbons (which could freeze at proposed storage temperatures of 210-270°R), reaction products of metal oxides from the walls of metal containers and, if used for seals, Teflon extractants or reaction products. Water and oxygen should be avoided.

3.2.1 B_2H_6 Doping Experiments

Stainless steel tubing and tankage, as well as fiber reinforced aluminum tanks, appeared to be attractive as construction material. Therefore, dopants selected were hydrocarbons, stainless steel oxides, and aluminum oxides. These were placed in aluminum and stainless steel tanks. These combinations should encompass most of the possible sources of contaminants which might interact to produce clogging. Dopants selected and storage tanks are listed below in Table 3-1. Teflon was chosen as a dopant because it is commonly used as valve seats.

Table 3-1. Storage Containers and Dopants For B_2H_6 Clogging Experiments

	Sample Number			
Condition	1	2	3	4
Dopant Tank	S.S. 347	6064-T6 alum.	6064-T6 alum.	6064-T6 alum.
Dopant	S.S. oxides 2.0 grams to pass 325 mesh screen	Alum. oxides S.S. oxides 1.0 gram each to pass 325 mesh screen	Low molecular weight hydrocarbons (Petroleum ether, 30 to 60°C, Baker Reagent Grade)	Finely divided Teflon 20/40 Mesh (T-5B duPont Molding Resin)

The doping apparatus, a tank which was attached to the flow system, is shown in Figure 3-1. This tank was loaded with diborane and dopant two weeks prior to the flow tests.

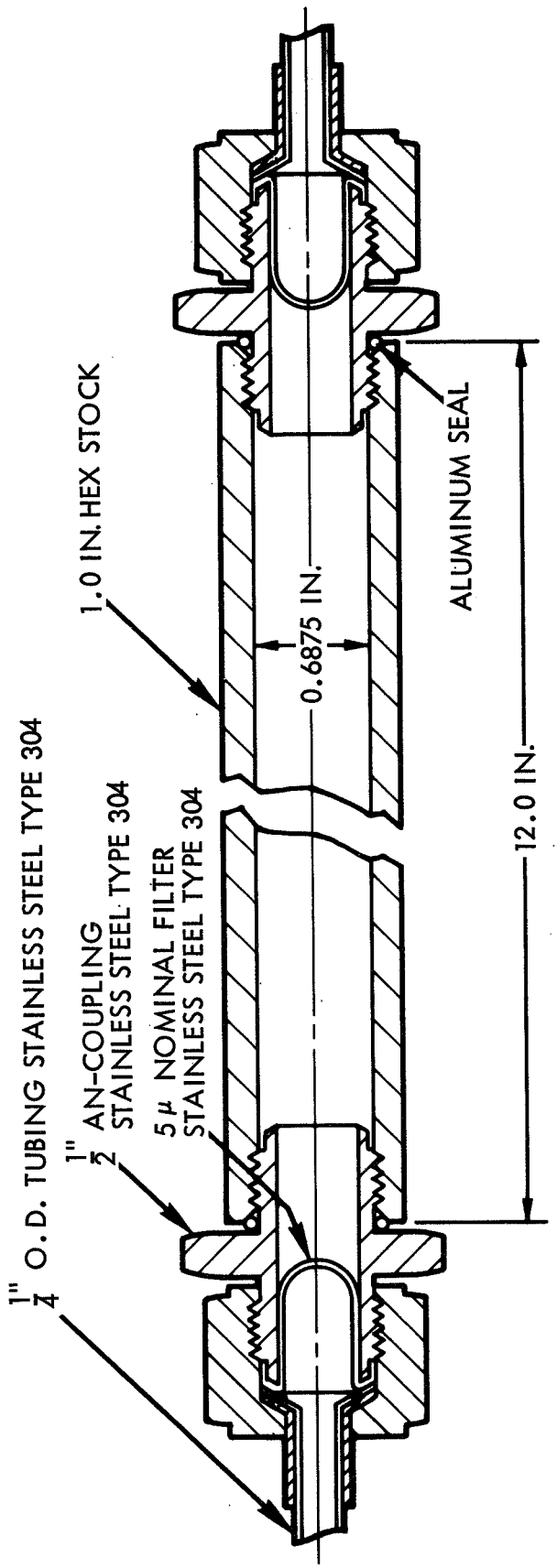

Figure 3-1. Doping Tank

The dopants and propellants were conditioned in the dopant tanks for two weeks at -108°F prior to testing. One to two grams of the dopant was added to approximately 40 cc of diborane. The doped diborane (20-40 cc) was introduced into the neat diborane (approximately 1000 cc) by flowing the entire sample of neat diborane through the dopant tank several times at -240°F. The large dopant particles were not permitted to enter the test orifice section because the dopant tank was capped at each end with 5 micron nominal rated filters.

The test cycle for doping the B_2H_6 propellant is outlined in Section 3.3.

3.2.2 Analysis of Diborane

Mass spectrometric analyses were performed on samples of diborane from both the diborane supply tank and the dopant conditioning tanks. These were sampled in a special way such as to cause a sample of the liquid phase to be delivered to the mass spectrometer inlet system. This was done by attaching the chilled tank with double valves to the spectrometer and inverting the tanks to deliver only liquid to the exhaust valve at the lower point on the tank. Enough liquid was admitted to the spectrometer for analyses. In addition, the diborane gas from the supply tank was also analyzed. The results of these analyses are shown in Table 3-2.

Table 3-2. B_2H_6 Analyses in Mole %

Constituent	B_2H_6 Tank Liq.	B_2H_6 Tank Gas	Sample 1 B_2H_6-D-SS	Sample 2 B_2H_6-D-Al	Sample 3 B_2H_6-D-HC	Sample 4 B_2H_6-D-TEF
Hydrogen	0.00	1.87	5.85	3.56	0.50	0.82
Argon	0.00	0.01	0.00	0.01	0.00	0.00
Hydrocarbon as pentane	0.02	0.05	0.01	0.00	5.99	0.00
Carbon dioxide	0.01	0.04	0.00	0.00	0.16	0.00
Methyl ethyl ether	0.08	0.00	0.00	0.00	0.00	2.11
Diborane	----------Balance----------					

The results show that Teflon introduced methyl-ethyl ether into the diborane sample. Also, samples 1 and 2 showed more hydrogen gas than the other samples. This probably comes from reaction of diborane with either water, hydroxides, or metal oxides to produce hydrogen. The 5.99% hydrocarbon in the B_2H_6 represents the added dopant, petroleum ether.

3.3 DIBORANE FLOW TEST PLAN

The test plan for the .010" diameter test orifice is presented in Table 3-3. The test procedure and operating conditions are outlined below.

1. Liquid diborane was transferred from the vendor's supply tank (at -100°F) to the test storage tank at -240°F. The liquid was filtered through a 7 micron rating filter to remove large particles.

2. The diborane was flowed from the storage tank through the 0.010" diameter test orifice to a bellows receiver tank held at the lower pressure P_1, P_2, P_3 and P_4 (Table 3-3).

3. Flow rate was determined by recording the position of a linear variable differential transformer (LVDT) attached to the receiver tank. Any change in flow rate was assessed by recording the change in EMF/time for the LVDT.

4. Temperature conditioning was achieved by heating the storage container to the required temperature and flowing the liquid through a heat exchanger located upstream of the test orifice.

5. The receiver tank pressure was maintained above the saturation vapor pressure to prevent a phase change (liquid to gas).

6. Parameters recorded were tank and receiver pressure and temperature, pressure drop and temperature drop across the test orifice.

Table 3-3. General Test Plan for the 0.010" Diameter Orifice Studies

Run No.	Supply Tank Temp.	Test Section Temp.	Inlet Pressure	Outlet* Pressure	Remarks
1	-240°F	-240°F	P_a	P_1	
2	↓	↓	↓	P_2	
3				P_3	
4				P_4	
5	-140°F ± 10	-240°F		P_1	Flow runs with temperature conditioning in the heat exchanger
6	↓	↓		P_2	
7				P_3	
8				P_4	
9	-60°F			P_1	Flow runs with temperature conditioning in the heat exchanger
10	↓	↓		P_2	
11				P_3	
12				P_4	
13	-140°F ± 10	140°F ± 10		P_1	
14	↓	↓		P_2	
15				P_3	
16				P_4	
17	-60°F	-60°F		P_1	
18	↓	↓		P_2	
19				P_3	
20				P_4	

P_a = Saturation Pressure + 200 psi

*$P_1 = P_{saturation} + 150$ psi

$P_2 = P_{saturation} + 100$ psi

$P_3 = P_{saturation} + 50$ psi

$P_4 = P_{saturation} + 5$ psi

The doped B_2H_6 propellant used in the flow runs is defined in Table 3-1. Table 3-4 is the dopant schedule used for the B_2H_6 flow runs. In all cases, the dopants were added to neat B_2H_6 indicated in the sample number defined in Table 3-1. The B_2H_6 propellants doped according to these samples were flowed through the different test sections varying flow velocity and temperature.

Table 3-4. B_2H_6 Propellant Run Schedule

Test Series	Test Section	Flash Distilled B_2H_6	Neat B_2H_6	B_2H_6 Sample Number (see Table 3-1) 1	2	3	4
1	0.010" Diameter Orifice	X					
2	0.010" Diameter Orifice		X				
3	0.010" Diameter Orifice		X	X			
4	0.010" Diameter Orifice		X	X	X		X
5	0.010" Diameter Orifice		X	X	X	X	X
6	0.020" Diameter Orifice		X				
7	2-10μ Filter		X				
8	2-10μ Filter		X	X	X	X	X
9	10μ Acquisition Screen		X				

3.4 FLOW BENCH CLEANING PROCEDURE

Cleaning of the individual components included a Freon and acetone wash. After assembly the flow bench was cleaned using the following procedure:

1. Flush with demineralized water
2. Isopropyl alcohol wash
3. Vacuum dry
4. Freon wash with trichlorotrifluoroethylene. (Precision Cleaning Agent, NASA Specification 273-A).
5. Vacuum dry.

During the above cleaning the water and alcohol solutions were cycled and filtered through a filter until no particles above 50μ were found. The filter used was a millipore filter rated at 15μ nominal. The filter residues were examined with a microscope during several runs of the wash cycle.

3.5 LEAKAGE CHECK PROCEDURES

Gaseous helium purge followed by helium pressure testing at 200 psig was the procedure used. Leakage was monitored using a helium leak detector.

3.6 DIBORANE FLOW TEST RESULTS

Forty selected diborane flow run test results are presented in Tables 3-5 to 3-14. The flow data showed no evidence of blockage or flow decay under all conditions of propellant conditioning for the three types of constrictions tested (orifices, filters, screen). After the tests were terminated and the test section removed, no observable residues were noted.

Several flow runs made with the 10μ acquisition screen are not reported. In these series flow decay was measured, but later was found due to a valve failure. The two flow runs reported in Table 3-5 were flowed with flash distilled diborane. This propellant was not planned in the original schedule and were special runs made to verify the apparent flow decay inadvertently due to a valve failure.

Variations noted in the average flow test data were, in most cases, under 10% for a single run. A few variations were noted approaching 20%. These larger variations were noted where the propellant was temperature conditioned. The large temperature transients may have caused changes in viscosity which effects flow, especially through the main lines. It was not possible to determine the exact cause of these flow anomalies. However, the anomalies showing a 20% variations were localized and no flow decay trends could be established.

The range of propellant velocities can be determined for the orifices tested. The diborane velocity using the 0.010" diameter orifice test section ranged from approximately 100 ft/sec to approximately 200 ft/sec through the 0.010" diameter hole. Using the data from run number one flowing neat B_2H_6 through the 0.010" diameter orifice (Table 3-6), the velocity is calculated to be about 100 ft/sec for the 2.0 cc/sec flowrate. Reynolds Number is about 10^5 based on a kinematic viscosity of 7×10^{-6} ft^2/sec. For the larger flowrates (4 cc/sec) Reynolds Number is about 2.0×10^5. Velocity is considerably lower for diborane using the other test sections; the flow area being much larger.

The data reported in Tables 3-5 to 3-14 are point data taken from strip chart recorders. These data were from representative runs covering the range of velocities, temperatures, and propellants flowed through the three types of constrictions in accordance with the flow schedule of Table 3-4. Visual examination of all the runs plotted on the strip chart were carefully made and no evidence of flow decay or clogging behavior could be found. Within the test conditions reported, it was concluded that diborane did not exhibit any flow anomalies within a 10% variation.

Table 3-5. Flow Test Data
Flash Distilled B_2H_6
.010" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		31	195	48	160	-240	-240	
	↓	2.14	60	↓	↓	↓	↓	↓	
		1.95	116.5						
		1.95	170.5						
		1.89	223.5						
		1.77	275.5						
		1.91	329.5						
		1.87	383						
		1.86	440						
2	200		17	195	185	0	-240	-240	
	↓	4.85	30	↓	↓	↓	↓	↓	
		3.93	58						
		3.90	85						
		3.64	112.5						
		3.48	139						
		3.57	167						
		3.66	224						

Table 3-6. Flow Test Data
Neat B_2H_6
.010" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		30.5	155	46	150	-140	-240	Temperature Conditioned
	↓	1.85	64	↓	45	↓	↓	↓	
		1.72	128		↓				
		2.0	190.5						
		1.63	252						
		1.60	313						
2	185		21.5	150	92	105	-140	-240	
	↓	2.76	44.0	↓	92	↓	↓	↓	
		2.54	87.5		88				
		2.50	129.5		88				
		2.40	171		86				
		2.40	212		86				
3	200		20	210	148	50	-140	-240	
	↓	3.26	39	↓	146	↓	↓	↓	
		2.90	77		144				
		2.92	113		142				
		2.70	150		142				
		2.80	185		142				
4	200		18.5	156	184	20	-140	-240	
	↓	3.26	37.5	↓	↓	20	↓	↓	
		3.06	73.5			15			
		3.38	104.5			↓			
		3.08	137						
		3.02	169.5						
		3.12	202.5						

Table 3-7. Flow Test Data
B_2H_6 Doped with Stainless Steel Oxides
.010" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		51	195	48	155	-240	-240	
	↓	2.1	102.5	↓	↓	↓	↓	↓	
		2.1	152.5						
		2.1	200.9						
		2.1	248.5						
		1.9	302.5						
2	200		25	192	48	165	-240	-240	
	↓	1.97	56.6	↓	↓	↓	↓	↓	
		2.1	108.8						
		2.08	159.2						
		2.04	208.4						
		2.06	256						
		1.86	311.4						

Table 3-8. Flow Test Data
B_2H_6 Doped with Stainless Steel and Aluminum Oxides
.010" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psi a	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		88	210	25	175	-240	-240	
	↓	1.25	176	↓	↓	↓	↓	↓	
		1.22	262						
		1.18	347						
		1.20	429						
		1.15	519						
		1.15	606						
2	200		37.4	190	97	105	-240	-240	
	↓	3.2	71.8	↓	↓	↓	↓	↓	
		3.2	105						
		3.2	136						
		3.0	169						
		3.0	203						
3	200		58	189	48	55	-240	-240	
	↓	2.1	110	↓	↓	↓	↓	↓	
		2.1	160.5						
		2.1	208.5						
		2.0	257.5						
		1.9	312						

Table 3-9. Flow Test Data
B_2H_6 Doped with Stainless Steel Oxides,
Aluminum Oxides and Teflon
.010" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	203		100.8	215	23	175	-240	-240	
	↓	1.33	184.4	↓	↓	↓	↓	↓	
		1.34	262.8						
		1.29	340.3						
		1.26	417.8						
		1.23	501.8						
		1.19	586.0						
2	203		66.0	215	46	155	-240	-240	
	↓	1.6	134.2	↓	↓	↓	↓	↓	
		2.0	185.0						
		2.0	235.4						
		2.0	284.2						
		2.0	336.4						
		1.9	388.0						
3	205		32.5	215	160	30	-240	-240	
	↓	3.44	64.5	↓	160	↓	↓	↓	
		3.62	93.5		166				
		3.45	122.5		166				
		3.50	150.5		166				
		3.42	180.5		166				
		3.36	210.7		165				

Table 3-10. Flow Test Data
B_2H_6 Doped with All Dopants
(see Table 3-1)
.010" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	205		18.5	210	146	50	-240	-240	
	↓	3.65	35.5	↓	↓	↓	↓	↓	
		3.54	66.5						
		3.56	96						
		3.58	124						
		3.18	153						
		3.32	184						
2	204		15.5	210	193	15	-240	-240	
	↓	4.2	30	↓	↓	↓	↓	↓	
		4.0	57.5						
		3.9	84.5						
		3.8	111						
		3.4	138.5						
		3.8	166						
		3.6	193.5						
3	203		19	210	142	50	-240	-240	
	↓	3.76	35.5	↓	↓	↓	↓	↓	
		3.44	67.5						
		3.50	97.5						
		3.30	127						
		3.10	156.5						
		3.22	188.5						
4	203		45	207	10	185	-240	-240	
	↓	.87	116	↓	↓	↓	↓	↓	
		.92	235						
		.92	349						
		.88	463						
		.81	584						

Figure 3-11. Flow Test Data
Neat B_2H_6
.020" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	270		13	270	130	125	-140	-140	
	↓	10	24	↓	↓	↓	↓	↓	
		11.6	33						
		10.9	42.2						
		10.6	51.4						
		12	60						
		10.9	69.2						
		10.6	79.2						
2	270		10.5	270	182	85	-140	-140	
	↓	12.5	19.3	↓	↓	↓	↓	↓	
		11.4	28.5						
		10.7	37.9						
		10.7	47.1						
		11	56.5						
		10.5	66						
		10.5	76.1						
3	200		12.6	180	175	10	-140	-140	
	↓	10.2	23.4	↓	↓	↓	↓	↓	
		10.1	33.8						
		10.0	43.8						
		10.2	53.4						
		10.4	63.3						
		10.1	73.2						
		9.8	84						

Table 3-11. Flow Test Data (Cont'd)
Neat B_2H_6
.020" Dia Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
4	260		17.4	212	62	175	-50	-170	Temperature Conditioning
	260	7.0	33.0	212	62	↓	↓	↓	↓
	260	7.5	47.0	212	62				
	260	8.0	59.5	212	62				
	280	8.3	71.2	235	78				
	280	8.4	83.4	235	78				
	280	8.0	95.8	235	76				
	280	7.5	109.0	235	76				
5	265		12.6	240	102	140	-50	-170	
	↓	10.6	24.3	↓	102	↓	↓	↓	
		10.6	35.4		102				
		9.5	46.0		102				
		9.3	56.6		101				
		10.0	66.8		101				
		10.0	77.8		100				
		9.6	88.8		100				
6	270		12	235	182	85	-50	-190	
	↓	10.2	22.8	↓	182	↓	↓	↓	
		11.9	31.6		181				
		10.2	40.5		180				
		10.9	49.5		179				
		12.1	58		178				
		11.1	67		176				
		11.5	76.2		174				

Table 3-12. Flow Test Data
Neat B_2H_6
2-10μ Filter

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		6.5	122	2	160	-240	-240	No Temperature Conditioning
	↓	17.5	12.8	↓	↓	↓	↓	↓	↓
		16.5	19.2						
		15.6	25.6						
		16.9	31.4						
2	150		5.6	97	3	105	-240	-240	
	↓	19.7	11.2	↓	↓	↓	↓	↓	
		19.1	16.7						
		18.2	22.2						
		18.1	27.6						
		20.0	32.7						
		18.4	38.2						
3	35		8.3	15	3	5	-240	-240	
	↓	13.4	16.5	↓	↓	↓	↓	↓	
		12.8	24.7						
		12.2	32.9						
		11.5	41.4						

Table 3-12. Flow Test Data (Cont'd)
Neat B_2H_6
2-10μ Filter

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
4	100		3.4	68	3	65	-180	-180	No Temperature Conditioning
	↓	17.7	9.6	↓	↓	↓	↓	↓	↓
		15.5	15.4						
		18.5	20.8						
		16.9	26.6						
		18.4	32.2						
		16.7	38.2						
		17.1	44.4						
5	90		6.0	50	4	30	-180	-180	
	↓	17.0	12.5	↓	↓	↓	↓	↓	
		19.1	18.0						
		23.2	22.3						
		20.8	27.0						
		21.0	31.9						
		20.0	36.9						
		20.8	42.0						
6	60		5	22	1	5	-180	-180	
	↓	22.0	10	↓		↓	↓	↓	
		20.2	15.2						
		20.8	20						
		19.6	25						
		22.4	29.6						
		20.8	34.4						
		19.6	39.8						

Table 3-13. Flow Test Data
B_2H_6 Doped with All Dopants
(see Table 3-1)
2-10μ Filter

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		2.5	115	4	100	-240	-240	
	↓	31.0	4.5	115	↓	105	↓	↓	
		31.5	8.0	118		↓			
		27.6	11.8	↓					
		28.4	15.5						
		28.0	19.0						
		29.5	22.5						
		28.6	26.0						
		30.2	29.5						
2	200		4.0	2.0	2	175	-240	-240	
	↓	13.8	8.5	↓	↓	↓	↓	↓	
		12.9	17.0						
		12.4	25.5						
		12.1	33.8						
		12.0	42.0						
		12.3	50.4						
		11.9	58.8						
3	NA	NA	NA	NA	NA	NA	-240	-240	Unable to Maintain Constant Supply Pressure
4	NA	NA	NA	NA	NA	NA	-240	-240	

Figure 3-13. Flow Test Data (Cont'd)
B_2H_6 Doped with All Dopants
(see Table 3-1)
2-10μ Filter

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psi a	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
5	100		7	66	2	55	-50	-150	Temperature conditioned
		18.3	13						
		21.0	18						
		18.2	23.5						
		19.6	28.5						
		17.2	34						
		16.7	40						
		21.1	45						
6	210		10.5	80	NA	55	-50	-150	
		44.2	13						
		35.0	16						
		33.5	19						
		39.0	21.5						
		41.5	24						
		41.6	26.5						
		39.3	29.2						
7	200		5	140	4	125	-50	-150	
		27.5	9						
		25.0	13.2						
		26.3	17						
		25.8	20.8						
		27.8	24.5						
		27	28.2						
		28	32						

Table 3-14. Flow Test Data
Neat B_2H_6
10μ Acquisition Screen

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		20.0	185	10	187	-240	-240	No temperature conditioning
	↓	2.66	62.8	↓	↓	↓	↓	↓	↓
		3.02	97.5						
		2.82	133.0						
		2.74	168.8						
		2.86	204.8						
		2.66	242.5						
		2.68	282.0						
2	203		12.6	190	21	165	-240	-240	
	↓	4.95	34.8	190	21	165	↓	↓	
		4.60	57.6	191	22	165			
		4.40	80.4	192	22	165			
		4.85	100.6	194	25	165			
		5.30	119.8	194	26	168			
		4.82	140.5	196	24	172			
		4.07	166.6	196	18	178			
3	156		14.0	160	40	100	-240	-240	
	↓	7.9	28.0	160	40	↓	↓	↓	
		7.6	41.8	161	40				
		7.2	55.8	161	40				
		7.5	68.8						
		7.7	82.2	161	40				
		6.9	96.8	162	40				
		7.1	111.8	163	38				

4.0 OXYGEN DIFLUORIDE FLOW STUDY

4.1 INTRODUCTION

The scope of this effort included testing the behavior of liquid oxygen difluoride propellant flowed through test sections made up of a .010 inch diameter orifice, a 2-10μ filter and a 10μ screen. The description of each test section is detailed under Section 2.0. Flow experiments were conducted in the temperature range of -180°F to -240°F and pressure range from 200 to 300 psig. Both neat and doped propellants were used with the .010 inch diameter orifice test section. Neat OF_2 was flowed through the filter and acquisition screens. Temperature conditioning of the OF_2 during flow was accomplished by passing the propellant through a heat exchanger located just upstream of the test section. The OF_2 was chilled from -180°F to -240°F for several experiments.

4.2 CHARACTERIZATION OF PROPELLANTS

Liquid oxygen difluoride was made by condensing the gas from the manufacturer's supply tank (Allied Chemical Corp.) without further characterization. Previous studies at TRW[1] showed liquid prepared by this method to be essentially free of Tyndall effect but could be caused to form a precipitate on partial evaporation of the liquid. This precipitate redissolved on addition of more OF_2 or could be caused to dissolve by heating the liquid.

The normal impurities CO_2, HF and SiF_4 were shown[1] to be of limited solubility in liquid at the proposed space environment storage range of -230°F. Under this program the liquid propellant was formed by condensing OF_2 with liquid nitrogen coolant at -322°F and showed very little Tyndall effect. The other impurity in OF_2 is CF_4 and did not form an insoluble liquid or solid phase at -322°F.

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(1) "Investigation of the Formation and Behavior of Clogging Material in Earth and Space Storable Propellants," TRW Systems Final Report No. 08113-6025-R000, Contract NAS 7-549, November 1969.

OF_2 can react with metal, M, or metal oxide, MO, surfaces according to Equations (1) and (2). If present as an impurity HF can react with metal surface according to Equation (3) and then be reformed according to Equation (4).

$$2\ OF_2 + 2M \longrightarrow 2MF_2 + O_2 \quad (1)$$

$$2\ F_2 + MO \longrightarrow MF_2 + O_2 \quad (2)$$

$$2\ HF + M \longrightarrow MF_2 + H_2 \quad (3)$$

$$2\ H_2 + OF_2 \longrightarrow 2HF + H_2O \quad (4)$$

If water is present on metal surfaces, HF could be formed according to Equation (5).

$$H_2O + OF_2 \longrightarrow 2HF + O_2$$

4.2.1 OF_2 Propellant Dopants

The dopants selected for the OF_2 propellant flow tests included the oxides of stainless steel and the oxides of aluminum.

The dopants mixed with neat OF_2 are shown in Table 4-1.

Table 4-1. Dopants for OF_2 Clogging Tests

	Sample Number	
Condition	1	2
Dopant Tankage	S. S. 347	6064-T6 Aluminum
Dopant	S. S. oxides 2.0 grams to pass 325 mesh screen	Aluminum oxides S. S. oxides 1.0 grams each to pass 325 mesh screen

The liquid OF_2 (condensed from the vendor's tank) was conditioned just prior to the flow tests by flowing the propellant through the dopants for a period of 15 to 20 minutes at -240°F several (8 to 10) times. The design of the dopant tank is described in Section 3.0.

4.3 TEST PLAN

The overall test plan for oxygen difluoride was similar for that of diborane. Details of the test plan are shown in Table 4-2.

The test sequence for the orifice, screen, filter and dopants was as follows:

1. The 12 experiments listed in Table 4-2 were performed with each type of test section.

2. The same series of 12 experiments were repeated with the 10 mil orifice using a doped oxygen difluoride supply. Dopant was stainless steel oxides 2.0 grams (which passed 325 mesh screen) in a stainless steel tank.

3. Repeat of (2) above with a mixture of Al_2O_3 and stainless steel oxides 2.0 grams total (which passed a 325 mesh screen) in an aluminum tank.

Dopant was supplied to the liquid oxygen difluoride in the same manner as with diborane.

Table 4-2. General Test Plan for OF_2
(Orifice, Screen and Filter)

Run No.	Supply Tank Temp.°F	Test Section Temp.°F	Inlet Pressure	Outlet Pressure	Remarks
1	-240 ± 10	-240 ± 10	P_a	P_1	normal experiment
2	↓	↓	↓	P_2	↓
3	↓	↓	↓	P_3	↓
4	↓	↓	↓	P_4	↓
5	-180 ± 10	↓	↓	P_1	*
6	↓	↓	↓	P_2	↓
7	↓	↓	↓	P_3	↓
8	↓	↓	↓	P_4	↓
9	↓	-180 ± 10	↓	P_1	normal experiment
10	↓	↓	↓	P_2	↓
11	↓	↓	↓	P_3	↓
12	↓	↓	↓	P_4	↓

P_a = saturation pressure + 200 psi
P_1 = $P_{saturation}$ pressure + 150 psi
P_2 = $P_{saturation}$ pressure + 100 psi
P_3 = $P_{saturation}$ pressure + 50 psi
P_4 = $P_{saturation}$ pressure + 5 psi

*These four (4) temperature conditioning experiments (Runs 5-8) in which LN_2 was used as coolant delivered OF_2 to the test section at about -240°F to -250°F depending upon the pressure.

4.4 CLEANING PROCEDURE

The flow tests with OF_2 followed the B_2H_6 testing. The cleaning procedure was designed to remove boron hydride and boric acid residues. This included:

1. Deionized water rinse
2. Isopropyl alcohol rinse
3. Vacuum dry
4. Freon cleaning with trichlorotrifluoroethylene (Precision Cleaning Agent, NASA Specification 273-A)
5. Vacuum dry
6. Passivation using a gas mixture of 80% fluorine and 20% oxygen.

4.5 LEAKAGE PROCEDURE

The leakage procedure included a gaseous helium purge followed by leakage check using a helium leak detector.

4.6 OF_2 FLOW TEST RESULTS

Tables 4-3 through 4-7 represent the results of the OF_2 flow test data. The flow data represents OF_2 flowed through the .010" diameter orifice, the 2-10μ filter and the 10μ acquisition screen. The propellant OF_2 was run isothermally as well as temperature conditioned by chilling the propellant prior to entering the test sections.

Four anomalies indicating flow had decreased were noted for the 10μ acquisition screen using neat OF_2 (See Run Nos. 5,6,7 and 8 of Table 4-7), temperature conditioned from -180°F to -240°F. The flow rate decreased more than 30% to about 60% of maximum flow rate. The flow decrease was also indicated by a large decrease in the ΔP across the test section. However, the upstream section inlet pressure decreased with decreasing ΔP. This indicated that the flow decrease was caused by conditions upstream in the system, possibly due to temperature conditioning at the heat exchanger.

A valve failure found during flow of the diborane propellant is also suspect. The valve failure was characterized by a slow closing of the valve seat and was due to a leak in the dome loaded actuator.

For those runs using the acquisition screen and when temperature remained constant, no observable flow anomalies occurred. For most of the OF_2 runs flow variations of 10% or more were common, believed resulting from variations in density due to temperature. In many cases, a 10% or more flow increase was noted, however, the ΔP was relatively unchanged, since approximately 10% flow variations also occurred when flowing diborane. It is believed these anomalies were inherent in the flow bench system and instrumentation.

Flow velocities ranged from 12 ft/sec to approximately 35 ft/sec through the .010" diameter orifice depending on the supply pressures and receiver pressure. Reynolds number for these velocities range from 5000 to approximately 10,000 indicating flow in the transition region. The Reynolds number was based on a kinematic viscosity of 2.0×10^{-6} ft^2/sec, which varies only slightly for the temperature range of -180°F to -240°F. Larger variations in density due to temperature are reported and range from 84.5 lb/ft^3 to 97.0 lb/ft^3, corresponding to -180°F and -240°F, respectively.

Table 4-3. Flow Test Data
Neat OF_2 .010" Dia. Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		220	162	44	150	-240	-240	
	↓	.22	468	165	44	↓	↓	↓	
		.22	925	160	43				
		.25	1350	170	42				
		.25	1720	170	42				
2	200		165	175	94	100	-240	-240	
	↓	.44	312	175	94	↓	↓	↓	
		.36	620	172	94				
		.43	850	172	94				
		.40	1110	172	92				
3	200		82	175	148	50	-240	-240	
	↓	.68	175	↓	140	↓	↓	↓	
		.63	335		144				
		.74	480		145				
		.60	635		137				
4	200		56	175	192	5	-240	-240	
	↓	1.28	105	175	190	↓	↓	↓	
		1.11	195	170	190				
		1.09	293	175	189				
		1.10	420	175	185				

Table 4-3. Flow Test Data
Neat OF_2 .010" Dia. Orifice (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
5	300		70	301	50	250	-180	-240	OF_2 Temperature Conditioned
	↓	.93	138	300	50	↓	↓	↓	↓
		1.02	236	304	49				
		1.08	335	306	49				
		1.0	474	307	48				
6	300		47	312	100	200	-180	-240	
	↓	1.44	90	314	95	↓	↓	↓	
		1.41	168	325	95				
		1.41	239	340	95				
		1.51	338	348	93				
7	300		40	304	149	150	-180	-240	
	↓	1.72	76	310	147	↓	↓	↓	
		1.64	143	321	146				
		1.64	204	321	146				
		1.27	322	330	142				
8	300		22	312	184	100	-180	-240	
	↓	3.26	41	320	183	↓	↓	↓	
		2.86	76	320	193				
		2.90	113	322	182				
		2.23	176	325	182				

Table 4-3. Flow Test Data
Neat OF_2 .010" Dia. Orifice (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
9	300		84	270	49	250	-180	-240	OF_2 Temperature Conditioned
	↓	.58	170	272	48	↓	↓	↓	↓
		.69	253	270	47				
		.55	340	270	48				
10	300		80	275	96	200	-180	-240	
	↓	.88	154	272	98	↓	↓	↓	
		.85	283	274	92				
		.86	400	275	90				
		1.04	455	275	89				
11	300		59	266	148	150	-180	-240	
	↓	1.26	108	267	148	↓	↓	↓	
		1.01	212	268	147				
		1.16	303	269	146				
		1.18	386	268	144				
12	300		45	258	186	100	-180	-240	
	↓	1.44	90	256	185	↓	↓	↓	
		1.28	172	255	181				
		1.37	245	258	180				
		1.44	318	258	178				

Table 4-4. Flow Test Data
OF_2 Doped with SS Oxides
.010" Orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		82	160	45	150	-240	-240	
	↓	.77	163	160	42	↓	↓	↓	
		.67	328	159	40				
		.74	463	158	39				
		.75	530	158	38				
2	200		52	158	98	100	-240	-240	
	↓	1.24	102	160	96	↓	↓	↓	
		1.22	192	160	90				
		1.17	282	160	92				
		1.28	321	160	90				
3	200		42.5	160	150	50	-240	-240	
	↓	1.51	83.5	160	150	↓	↓	↓	
		1.33	166.5	160	150				
		1.51	235.5	159	140				
		1.56	267.5	158	148				
4	200		38	195	NA	5	-240	-240	
	↓	1.64	76	192	↓	↓	↓	↓	
		1.34	158	190					
		1.47	192	188					
		1.51	225	186					
5	300	1.0	70	210	60	250	-180	-240	OF_2 Temperature Conditioned
	↓	.86	132.5	212	53	↓	↓	↓	↓
		.98	260	214	53				
		.89	367	215	48				
			474	216	46				

Table 4-4. Flow Test Data
OF_2 Doped with SS Oxides
.010" Dia Orifice (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
6	300		51.5	216	94	200	-180	-240	OF_2 Temperature Conditioned
	↓	1.19	103.5	218	92	↓	↓	↓	↓
		1.18	197	218	90				
		1.18	282	218	89				
		1.2	323.5	218	94				
7	300		44	214	142	150	-180	-240	
	↓	1.41	88	212	138	↓	↓	↓	
		1.43	165	214	135				
		1.58	231.5	216	130				
		1.50	265	218	130				
8	300		37	212	188	105	-180	-240	
	↓	1.70	73.5	↓	184	↓	↓	↓	
		1.63	141		183				
		1.81	199		180				
		1.85	226		178				
9	300		61	218	52	250	-180	-180	
	↓	1.04	120.5	220	50	↓	↓	↓	
		1.00	230	222	49				
		1.00	330.5	223	48				
		1.17	419	226	46				
		1.01	510	226	43				

Table 4-4. Flow Test Data
OF_2 Doped with SS Oxides
.010" Dia Orifice (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
10	300		48.5	228	84	200	-180	-180	
	↓	1.39	92	235	95	↓	↓	↓	
		1.37	173.5	235	92				
		1.34	252	238	90				
		1.46	320.5	239	89				
		1.25	358	239	88				
		1.16	400	240	87				
11	300		46	234	150	150	-180	-180	
	↓	1.67	83	240	148	↓	↓	↓	
		1.61	151.5	242	146				
		1.63	216	244	142				
		1.78	272	244	141				
		1.50	302	244	140				
		1.30	338	244	140				
		1.15	364	244	140				
12	300		55	236	162	140	-180	-180	
	↓	1.23	106	238	158	↓	↓	↓	
		1.39	142	240	158				
		1.52	211	242	155				
		1.28	269	244	152				
		1.41	334	244	152				

Table 4-5. Flow Test Data
OF_2 Doped with Aluminum and Stainless Steel
Oxides .010" Dia. orifice

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		76	NA	44	150	-240	-240	
	↓	.84	150	↓	42	↓	↓	↓	
		.82	285		44				
		1.05	380		42				
		.62	626		42				
2	200		51	NA	96	100	-240	-240	
	↓	1.24	101	↓	94	↓	↓	↓	
		1.12	200		91				
		1.12	289		90				
		1.20	414		88				
3	200		42	NA	152	50	-240	-240	
	↓	1.55	82	↓	152	↓	↓	↓	
		1.24	162		148				
		1.26	240		146				
		1.48	341		142				
4	200		40	NA	180	5	-240	-240	
	↓	1.55	80	↓	100	↓	↓	↓	
		1.38	160		178				
		1.56	224		172				
		1.41	330		162				

Table 4-5. Flow Test Data
OF_2 Doped with Aluminum and Stainless Steel Oxides .010" Dia. orifice (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
5	300		40	220	182	105	-180	-240	OF_2 Temperature Conditioned
	↓	1.63	78	222	180	↓	↓	↓	↓
		2.10	108	226	173				
		1.56	146	226	172				
		1.76	163	230	172				
6	300		43	222	140	150	-180	-240	
	↓	1.59	82	226	140	↓	↓	↓	
		1.36	163	232	138				
		1.36	233	234	138				
		1.55	330	236	135				
7	300		108	227	94	200	-180	-240	
	↓	1.10	208	232	92	↓	↓	↓	
		1.22	290	232	91				
		1.19	416	234	89				
8	300		76	215	62	150	-180	-240	
	↓	0.9	145	190	60	↓	↓	↓	
		1.0	255	194	58				
		0.9	366	196	56				
		1.03	526	200	50				

Table 4-5. Flow Test Data
OF_2 Doped with Aluminum and Stainless Steel Oxides, .010" Dia. Orifice (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
9	300		108	247	58	250	-180	-180	
	↓	.95	224	251	58	↓	↓	↓	
		.97	430	252	57				
		.79	547	256	55				
10	300		58	248	95	200	-180	-180	
	↓	.94	175	250	93	↓	↓	↓	
		1.20	345	251	94				
		1.19	465	255	92				
11	300		76	245	150	150	-180	-180	
	↓	.84	150	250	148	↓	↓	↓	
		.91	250	252	148				
		1.2	330	256	142				
12	300		50	244	182	105	-180	-180	
	↓	1.64	68	244	182	↓	↓	↓	
		2.10	120	245	179				
		1.67	180	245	178				
		1.75	240	245	178				
		2.2	280	244	178				

Table 4-6. Flow Test Data
Neat OF_2, 2-10 Micron Filter

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		5.7	122	4	150	-240	-240	
	↓	10.9	12	125	4	↓	↓	↓	
		10.0	23	126	4				
		9.6	33.8	127	4				
		10.0	43.8	128	4				
2	200		3.5	96	7	100	-240	-240	
	↓	17.6	7	97	7	↓	↓	↓	
		15.7	14	98	7				
		17.5	20	98	7				
		16.7	26	98	7				
3	200					50	-240	-240	
	↓	25	2.4	60	8	↓	↓	↓	
		20.3	5.4	63	8				
		18.6	11.3	66	8				
		19.8	16.6	69	8				
		20.0	21.6	74	8				
4	200		2.5	36	14	5	-240	-240	
	↓	24.8	5	38	13	↓	↓	↓	
		22	10	40	13				
		25	14	43	11				
		26	18	46	12				

Table 4-6. Flow Test Data
Neat OF_2, 2-10 Micron Filter (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
5	300		4.7	210	7	235	-180	-180	
	↓	13.2	9.4	210	7	↓	↓	↓	
		12.8	18.0	210	7				
		13.1	26.0	210	7				
		13.1	33.6	210	7				
6	300		4.1	200	3	235	-180	-180	
	↓	15.1	8.2	200	3	↓	↓	↓	
		15.3	15.4	200	3				
		14.2	22.8	200	3				
		16.1	29	200	3				
		14.0	36.0	200	3				
7	300		3.5	178	8	197	-180	-180	
	↓	20.0	6.6	178	8	↓	↓	↓	
		17.8	12.8	178	8				
		20.0	18.0	178	8				
		18.9	23.2	178	8				
		16.3	29.3	178	8				
8	300		3	135	10	150	-180	-180	
	↓	24.8	5.5	137	10	↓	↓	↓	
		22	10.5	140	10				
		20	15.5	140	10				
		21	20.5	140	10				
		23	24.5	140	10				

Table 4-6. Flow Test Data
Neat OF_2, 2-10 Micron Filter (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
9	300		3.0	108	17	105	-180	-180	
	↓	31.0	5.0	108	15	↓	↓	↓	
		27.5	9.0	108	13.5				
		26.3	13.0	108	13				
		33.0	16.0	108	12.5				
		25.8	20.2	108	12.5				
10	300		4	187	3	230	-180	-240	OF_2 Temperature Conditioned
	↓	15.5	8	187	3	↓	↓	↓	↓
		13.8	16	187	3				
		14.3	23	187	3				
		13.2	31	187	3				

Table 4-7. Flow Test Data
Neat OF_2, 10 Micron Acquisition Screen

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
1	200		53	156	45	150	-240	-240	
	↓	1.19	105	162	41	150	↓	↓	
		1.05	210	160	40	150			
		1.02	308	162	40	150			
		1.12	441	162	40	150			
2	200		33						
	↓	1.67	70	165	94	100	-240	-240	
		1.57	140	165	91	100	↓	↓	
		1.85	194	164	90	100			
		1.95	248	163	89	100			
		1.55	277	162	88	100			
3	200		25	165	139	50	-240	-240	
	↓	2.48	50	160	132	50	↓	↓	
		2.75	90	164	131	50			
		2.0	140	164	130	50			
		2.7	179	164	130	50			
		2.4	198			50			
4	200		20	168	182	5	-240	-240	
	↓	3.1	40	168	181	5	↓	↓	
		2.9	78	167	181	5			
		3.1	110	165	180	5			
		3.6	139	165	180	5			
		2.8	155	165	179	5			

Table 4-7. Flow Test Data
Neat OF_2, 10 Micron Acquisition Screen (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
5	300		20	250	51	250	-180	-240	Temperature Conditioned
	↓	3.26	39	250	50	↓	↓	↓	↓
		2.56	82	244	40				
		2.0	132	235	28				
		1.75	192	230	22				
		1.32	225	225	20				
6	300		16	246	70	200	-180	-240	
	↓	3.87	32	245	68	↓	↓	↓	
		3.24	66	238	58				
		3.40	97	230	49				
		2.94	131	208	34				
		1.95	154	204	29				
7	300		12	224	108	150	-180	-240	
	↓	5.64	23	222	104	↓	↓	↓	
		4.4	48	218	95				
		4.3	71	208	88				
		4.8	93	204	72				
		3.75	105	192	70				
7A	300		11	246	111	150	-180	-240	
	↓	5.64	22	228	102	↓	↓	↓	
		4.6	46	222	94				
		4.2	70	216	86				
		4.6	93	208	88				
		3.5	106	204	72				

Table 4-7. Flow Test Data
Neat OF_2, 10 Micron Acquisition Screen (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
8	300		10			250	-180	-180	
	↓	5.9	20.5	254	45	↓	↓	↓	
		6.1	38.5	250	44				
		5.7	56	260	43				
		6.5	72	262	42				
		5.3	89.5	264	40				
9	300		7.5	254	81	200	-180	-180	
	↓	8.9	14.5	256	80	↓	↓	↓	
		8.2	28	260	79				
		8.3	40	262	78				
		8.7	52	262	77				
		7.7	64	262	77				
10	300		7	242		150	-180	-180	
	↓	9.5	13.5	248	120	↓	↓	↓	
		11.5	23	248	118				
		10.5	32.5	249	116				
		11.0	42	250	114				
		8.8	52.5	250	112				
11	300		5	235	160	105	-180	-180	
	↓	15.5	9	246	158	↓	↓	↓	
		15.7	16	248	155				
		16.7	22	249	155				
		17.5	28	250	154				
		18.4	33	254	154				

Table 4-7. Flow Test Data
Neat OF_2, 10 Micron Acquisition Screen (Cont.)

Run No.	Supply Pressure psig	Average Flow cc/sec	Datum Point Time sec	Test Section Pressure psia	Test Section Differ. Pressure psid	Receiver Pressure psig	Storage Temp. °F	Test Section Temp. °F	Remarks
12	300		10	210	140	105	-180	-240	Temperature Conditioned
	↓	5.9	20.5	206	133	↓	↓	↓	↓
		5.5	40.5	200	127				
		5.2	60	195	120				
		5.8	78	190	111				
		5.0	87	187	107				

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